MANUAL FOR CHEMISTRY PRACTICALS FOR ICD CLASSES FIRST SEMESTER

Subject Code: CY-111 (Chemistry-I)

CHEMISTRY LAB

DEPARTMENT OF CHEMISTRY
SLIET, LONGOWAL (DEEMED UNIVERSITY)
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SYLLABUS

L	T	P	Cr
4	0	2	5

LIST OF PRACTICALS

1. To prepare the standard solution of oxalic acid or potassium dichromate.
2. To determine the strength of given HCl solution by titration against NaOH solution using Phenolphthalein indicator.
3. To determine the total hardness of water sample in terms of $CaCO_3$ by EDTA titration method using Eriochrome Black-T indicator.
4. To determine the pH of given sample.
5. To analyse inorganic salt for acidic and basic radicals among the following.
   A. Basic Radicals:
   NH_4^+, Pb^{++}, Cu^{++}, Bi^{+++}, Cd^{++}, As^{+++}, Sb^{+++}, Sn^{++}, Al^{+++}, Fe^{+++}, Cr^{+++}, Mn^{++}, Zn^{++}, Co^{++}, Ni^{++}, Ba^{++}, Sr^{++}, Ca^{++}, Mg^{++}
   B. Acid Radicals:
   CO_3^{--}, S^{--}, SO_3^{--}, CH_3COO^-, NO_2^-, NO_3^- , Cl^-, Br^- , I^- , SO_4^{--}

CONTENTS

Instructions to the Students

- Keep work area neat and free of any unnecessary objects.
- Never pour chemical waste into the sink drains or wastebaskets.
- Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
- Wear disposable gloves, as provided in the laboratory, when handling hazardous materials.
- Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
- Wear shoes that adequately cover the whole foot.
- Keep your hands away from your face, eyes, mouth, and body while using chemicals.
- In case of an emergency or accident, follow the established emergency plan as explained by the teacher and evacuate the building via the nearest exit.
- Always use a spatula to remove a solid reagent from a container.
- Use a hot water bath to heat flammable liquids. Never heat directly with a flame.
- Add concentrated acid to water slowly. Never add water to a concentrated acid.
- Never place the container directly under your nose and inhale the vapors.
- Never mix or use chemicals not called for in the laboratory exercise.
- Clean up all spills properly and promptly as instructed by the teacher.
- Thoroughly clean your laboratory work space at the end of the laboratory session. Do not block the sink drains.

Experiment: 1

Aim: To prepare the standard $\frac{N}{10}$ oxalic acid solution.

Requirements:

Apparatus: measuring flask, funnel, watch glass and wash bottle.

Chemicals: Oxalic acid, distilled water

Procedure

Weigh accurately 1.575 g Oxalic acid on dry watch glass and transfer it to a volumetric flask (250 ml) with the help of funnel, half-filled with distilled water. Shake well until whole the solid dissolve and make the volume up to the mark with the help of dropper. Label it as $\frac{N}{10}$ oxalic acid solution.

Note: If anhydrous oxalic acid is available then dissolve 4.5 g of the acid in one litre of distilled water to get 0.1 N oxalic acid solution.

Calculations:

$$\text{Normality} = \frac{\text{weight of solute (gm)}}{\text{equivalent weight of solute}} \times \frac{1000}{\text{volume of solution (ml)}}$$

$$\text{Eq. wt. of Oxalic acid} = \frac{\text{Molecular weight}}{\text{Basicity}} = \frac{126}{2} = 63$$

Experiment: 2

Aim*:* Determination of strength of NaOH solution by titrating against $\frac{N}{20}$ HCl solution.

Requirements: Burette, pipette, titration flask, beakers and funnel.

Chemicals: Phenolphthalein, NaOH solution and standard solution of $\frac{N}{20}$ HCl.

Indicator*:* Phenolphthalein.

End point: Pink color to colorless.

Chemical reaction:

$NaOH + HCl \rightarrow NaCl + H_2O$

Theory:

It is acid-base titration, in which one gram equivalent NaOH is completely neutralized by acid and enthalpy change is called as enthalpy of neutralization. The normality of NaOH is determined by titrating it against N/20 HCl. The normality is then multiplied by equivalent weight to get the strength of NaOH solution. Strength is written in gm/litre.

Procedure:

Wash the whole glass wares with the water. Rinse the funnel and burette with N/20 HCl. Fill the burette with N/20 HCl by means of funnel. Note the initial reading of the burette.Rinse the pipette with NaOH solution. 20ml of NaOH solution is taken in titration flask and add 2 to 3 drops of phenolphathalein. The color of the solution turns to pink. Run the HCl solution from the burette into the titration flask drop wise. Give the swirling motion to the titration flask after each addition. Continue adding acid with a last single drop, the pink color just disappear. Note the final reading of the burette. Repeat the above experiments to get concordant reading.

Observations:

Serial no.	Initial reading	Final reading	Volume of HCl used (ml)
1.			
2.			

Concordant reading = ………….. mL

Apply Normality equation

$$N_1 \times V_1 = N_2 \times V_2$$
(NaOH) (HCl)

$$N_1 = (N_2 \times V_2) / V_1$$
$$= A \text{ (say)}$$

Strength of given NaOH solution =Normality × Equivalent Weight

$$= A \times 40$$
$$= B \text{ g/L}$$

Result: The strength of the given NaOH is ………… g/L.

Experiment: 3

Aim: To determine the total hardness in the water by using N/20 EDTA solution.

Requirements:

Apparatus: Burette, pipette, conical flask, funnel, beaker, stand.

Chemicals: EDTA solution, water solution, Eriochrome black T, Buffer solution

Indicator: Eriochrome black T

End Point: Light pink to blue

Theory:

Hard water is that which has high mineral content. Hard Water contains bicarbonates, chlorides, sulphates of calcium & magnesium. When hard water treated with soap it gets precipitated in the form of insoluble salts of Ca & Mg. Hardness of water is a measure of total concentration of Ca & Mg ions expressed as $CaCO_3$. There are two types of hardness:

Temporary hardness: It is due to presence of bicarbonates of Ca & Mg. It can be easily removed by physical method i.e. boiling.

Permanent hardness: It is due to presence of chlorides & sulphates of Ca & Mg. It cannot be removed by boiling.

Procedure:

1. Rinse and fill the burette with N/20 EDTA solution and noted the initial reading of the burette.
2. Pipette out 20ml water sample to the conical flask.
3. Add 2 ml of buffer solution (basic buffer) and two drops of Eriochrome black T.
4. Titrated the contents of conical flask against EDTA solution with continuous shaking till the end point appeared.
5. Repeated the procedure to get concordant reading.

Formula Used:

Total hardness = volume of EDTAsolution × normality × 50 × 1000/ volume of sample taken

Observations:

S.No.	Initial Reading	Final Reading	Volume Of EDTA Used (mL)
1.			
2.			

3.			

Concordant reading = ………………………..mL

Total hardness = ………………………………..mg /L

Result: Calcium hardness as $CaCO_3$equivalent (mg/l) in given water sample was found to be ………………………mg/L as $CaCO_3$ equivalent .

Experiment: 4

Aim: To determine the pH of given samples.

Requirements:

Apparatus: Beaker, glass rod, dropper and pH paper.

Chemicals: dil. HCl, dil. CH_3COOH, NaOH, $NaHCO_3$, and distilled water.

Procedure:

1. Take a small amount ofdifferent solutions in test tubes marked 1,2,3…..
2. Dip a small piece of pH paper in test tube 1 and note the color of the pH paper. Match the color of the pH scale and determine the pH of the solution and recorded in table.
3. Similarly determine the pH of other solutions and record your observation.

Observations:

Sr. No.	Solution	pH	Nature of solution (acidic, basic & neutral)
1.	dil HCl		
2.	dil CH_3COOH		
3.	water		
4.	NaOH		
5.	$NaHCO_3$		

Result: On the basis of observation the pH of dil. HCl, dil. CH_3COOH, NaOH, $NaHCO_3$, and distilled water are ---------------------- respectively.

Experiment : 5

Aim*:* To analyze inorganic salt for acid radicals.

CATIONS: Pb^{2+},Hg^{2+},Cu^{2+},Cd^{2+}, Ag^{+}, Fe^{2+}, Fe^{3+}, Al^{3+}, Zn^{2+}, Mn^{2+}, Co^{2+}, Sr^{2+}, Ba^{2+},Mg^{2+}, NH_4^{+}

ANIONS: CO_3^{2-}, S^{2-}, SO_3^{2-}, SO_4^{2-}, NO_2^{-}, NO_3^{-}, Cl^{-}, Br^{-}, PO_4^{3-}, CH_3COO^{-}

Preliminary Tests

1. Note the state (amorphous or crystalline) and color of salt.

2. Test the solubility of the salt in the following solutions:

    i. Water (cold and hot)

    ii. Dilute HCl (cold and hot)

    iii. Dilute HNO_3(cold and hot)

IDENTIFICATION OF ACID RADICALS (ANIONS)

This part is the classified into three groups:

<u>FIRST GROUP OF ACID RADICALS</u>

The acid radicals involved in this group are carbonate (CO_3^{2-}), Sulphide (S^{2-}), Sulphite(SO_3^{2-}), Thiosulphate ($S_2O_3^{2-}$) and nitrite(NO_2^{-}). The group reagent is **dilute H_2SO_4acid.**

EXPERIMENT	OBSERVATION	INFERENCE
Salt + dil. H_2SO_4	effervescence or evolution of gases	1st group of acid radicals is present.
	(a) Colorless, odorless gas turns lime water milky	The acid radical may be CO_3^{2-}or HCO_3^{-}
	(b) Colorless with rotten eggs smell and turnslead acetate paper black.	Sulphide(S^{2-}) confirmed.
	(c) Colorless gas with suffocating smell. Heat and pass the gas through acidified $K_2Cr_2O_7$solution. The solution turns green	SO_3^{2-} confirmed.
	(d) Colorless gas followed by brown gas and it turns starch iodide paper blue	NO_2^{-} confirmed.
	(e) Colorless gas with the smell of vinegar.	The acid radical may

		be CH_3COO^-

Chemical reactions involved in dilute H_2SO_4 acid test:

Test for Carbonate ion (CO_3^{-2}):

$Na_2CO_3 + H_2SO_4 \rightarrow Na_2SO_4 + H_2O + CO_2\uparrow$
$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$

Test for Sulphide ion [S^{-2}]

$Na_2S + H_2SO_4 \rightarrow Na_2SO_4 + H_2S\uparrow$
$(CH_3COO)_2Pb + H_2S \rightarrow \underset{\text{black}}{PbS\downarrow} + 2CH_3COONa$

Test for Sulphite ion [SO_2^{3-}]

$K_2Cr_2O_7 + H_2SO_4 + 3SO_2 \rightarrow K_2SO_4 + \underset{\text{Chromium(III) sulphate (green)}}{Cr_2(SO_4)_3} + H_2O$

Test for Nitrite ion [NO_2^-]

$NO_2^- + CH_3COOH \rightarrow HNO_2 + CH_3COO^-$
$2HNO_2 + 2KI + 2CH_3COOH \rightarrow 2CH_3COOK + 2H_2O + 2NO + I_2$
I_2 + Starch $\rightarrow$ Blue complex

Test for Acetate ion [CH_3COO^-]

$2\ CH_3COONa + H_2SO_4 \rightarrow Na_2SO_4 + 2\ CH_3COOH$
$CH_3COOH + C_2H_5OH \rightarrow \underset{\text{Ethyl acetate (fruity odour)}}{CH_3COOC_2H_5} + H_2O$

Tests to distinguish between CO_3^{2-} and HCO_3^-

EXPERIMENT	OBSERVATION	INFERENCE
Salt + water, boil and pass the gasthrough lime water	(a) Lime water does not turn milky.	CO_3^{2-} confirmed
	(b) Lime water turns milky.	HCO_3^- confirmed.

*****Like CO_2 sulphur dioxide also turns lime water milky. But CO_2 is odourless gas and SO_2 has a characteristic smell.

Confirmation test for Acetate

EXPERIMENT	OBSERVATION	INFERENCE
Salt solution + Neutral $FeCl_3$ sol.	Appearance of blood red colour.	Acetate confirmed

$6\ CH_3COO^- + 3Fe^{3+} + 2H_2O \rightarrow [Fe_3(OH)_2(CH_3COO)_6]^+ + 2H^+$

$[Fe_3(OH)_2(CH_3COO)_6]^+ + 4H_2O \rightarrow 3[Fe(OH)_2(CH_3COO)] + 3CH_3COOH + H^+$

Iron(III)dihydroxyacetate (Brown-red ppt)

SECOND GROUP OF ACID RADICALS

The acid radicals involved in this group are Cl^-, Br^- and NO_3^-. The group reagent is **conc. sulphuric acid (H_2SO_4).**

EXPERIMENT	OBSERVATION	INFERENCE
Salt +Conc. H_2SO_4 and heatif necessary	Effervescence with colorless or colored gases	2[nd]group Acid radical is present
	(a) Colorless gas with a pungent smell and gives dense white fumes when a glass rod dipped in ammonium hydroxide (NH_4OH) is exposed.	Acid radical may be Cl^-
	(b) Brown gas	Acid radical may be Br^-
	(c)Violet vapours	Acid radical may be I^-
	(d) Light brown gas and brown gas with pieces of copper turnings and the solution turns blue in the test tube.	Acid radical may be NO_3^-

Confirmation test for Chloride

EXPERIMENT	OBSERVATION	INFERENCE
(a) Chromyl Chloride test: Salt + few $K_2Cr_2O_7$ crystals + conc. H_2SO_4 and heat. (b) Pass the vapors through the test tube	Red vapours are obtained. The solution turns yellow.	Chloride confirmed

which contains NaOH solution. (c) To this yellow solution, add dilute CH_3COOH and lead acetate solution.	Yellow ppt. is formed	
Silver Nitrate test: Salt solution + $AgNO_3$ solution + dilute HNO_3	White ppt. is formed which is soluble in NH_4OH.	Chloride confirmed.

Test for Chloride ion [Cl^-]

Chromyl – Chloride test:

$$4NaCl + K_2Cr_2O_7 + 6H_2SO_4 \rightarrow 2KHSO_4 + \underset{\text{(Chromylchloride)}}{2CrO_2Cl_2} + 4NaHSO_4 + 3H_2O$$

$$CrO_2Cl_2 + 4NaOH \rightarrow Na_2CrO_4 + 2NaCl + 2H_2O$$

$$(CH_3COO)_2Pb + Na_2CrO_4 \rightarrow \underset{\substack{\text{Lead chromate} \\ \text{(Yellow precipitate)}}}{PbCrO_4\downarrow} + 2CH_3COONa$$

(a) Silver Nitrate test:

$$NaCl + AgNO_3 \rightarrow NaNO_3 + \underset{\text{Silver chloride (White precipitate)}}{AgCl\downarrow}$$

Confirmation test for Bromide

EXPERIMENT	OBSERVATION	INFERENCE
(b)Silver Nitrate test: Salt solution + $AgNO_3$ solution + dilute HNO_3	A light yellow ppt. slightly soluble in NH_4OH is obtained	Bromide confirmed.

Test for Bromide ion (Br^-)

(a) $NaBr + AgNO_3 \rightarrow NaNO_3 + AgBr\downarrow$

(Silver bromide) pale yellow ppt

Confirmation test for Iodide

EXPERIMENT	OBSERVATION	INFERENCE
Silver Nitrate test: Salt solution + $AgNO_3$solution + dilute HNO_3	A yellow ppt. insoluble in NH_4OH	Iodide confirmed.

Test for Iodide ion (I^-)

$NaI + AgNO_3 \rightarrow AgI \downarrow + NaNO_3$
silver iodide(Yellow precipitate)

Confirmation test for Nitrate

EXPERIMENT	OBSERVATION	INFERENCE
Brown ring test: Strong solution of the substance + 2-3 drops of conc. H_2SO_4, and cool. Add freshly prepared $FeSO_4$ solution along the sides of the test tube.	A brown ring is formed at the junction of twoliquids.	Nitrate confirmed

Test for Nitrate ion [NO_3^-]

$NaNO_3 + H_2SO_4 \rightarrow NaHSO_4 + HNO_3$
$6\ FeSO_4 + 3H_2SO_4 + 2HNO_3 \rightarrow 3Fe_2\ (SO_4)_3 + 4H_2O + 2NO$
$FeSO_4 + NO + 5\ H_2O \rightarrow [Fe(NO)(H_2O)_5]SO_4$
Nitroso ferrous sulphate(Brown)

<u>THIRD GROUP OF ACID RADICALS</u>

The basic radicals involved in this group are $SO_4^{2-}$and PO_4^{3-}.

EXPERIMENT	OBSERVATION	INFERENCE
Test for sulphate: Aqueous solution of salt + dil. HCl + $BaCl_2$solution.	A white ppt. insoluble in diluteHCl is obtained	Sulphate (SO_4^{2-}) confirmed

Test of Sulphate ions [SO_4^{2-}]

$Na_2SO_4 + BaCl_2 \rightarrow \quad BaSO_4 \downarrow + 2NaCl$
Barium sulphate
(White precipitate)

Experiment: 6

Aim*:* To analyze inorganic salt for basic radicals.

Basic radicals are classified into six groups. They are mentioned as below:

GROUP	RADICALS	GROUP REAGENTS
zero	NH_4^+	none
I	Pb^{2+}	Dilute Hydrochloric acid (HCl)
II	Pb^{2+}, Cu^{2+}, As^{3+}	Dilute HCl + H_2S gas.
III	Al^{3+},Fe^{3+}	NH_4Cl (s) + NH_4OH
IV	Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}	NH_4Cl (s) + NH_4OH + H_2S gas
V	Ca^{2+}, Sr^{2+}, Ba^{2+}	$NH_4Cl(s)$ + NH_4OH + $(NH_4)_2CO_3$
VI	Mg^{2+}	none

Wet Tests for Identification of Cations

The cations indicated by the preliminary tests given above are confirmed bysystematic analysis given below.The first essential step is to prepare a clear and transparent solution of thesalt. This is called **original solution.** It is prepared as follows:

Preparation of Original Solution (O.S.)

To prepare the original solution, following steps are followed one after the otherin a systematic order. In case the salt does not dissolve in a particular solventeven on heating, try the next solvent.

The following solvents are tried:

1. Take a little amount of the salt in a clean boiling tube and add a few mL ofdistilled water and shake it. If the salt does not dissolved, heat the contentof the boiling tube till the salt completely dissolves.

2. If the salt is insoluble in water as detailed above, take fresh salt in a cleanboiling tube and add a few mL of dil. HCl to it. If the salt is insoluble incold, heat the boiling tube till the salt is completely dissolved.

3. If the salt does not dissolve either in water or in dilute HCl even on heating,try to dissolve it in a few mL of conc. HCl by heating.

4. If salt does not dissolve in conc. HCl, then dissolve it in dilute nitric acid.

5. If salt does not dissolve even in nitric acid then a mixture of conc. HCl andconc. HNO_3 in the ratio 3:1 is tried. This mixture is called aqua-regia. A saltnot soluble in aqua-regia is considered to be an insoluble salt

ZERO GROUP OF BASIC RADICALS

Test for NH_4^+ Radicals: This test is carried out before starting the analysis for the basic radicals.

EXPERIMENT	OBSERVATION	INFERENCEE
(a) Salt + NaOH solution and heat it. Expose a glass rod dipped in conc. HCl to the gas and moist red litmus paper.	Colorless gas with a pungent smell is obtained. Dense white fumes and moistred litmus paper turns blue	NH_4^+may be present in the salt
(b) Original solution + Nesseler's reagent.	Brown solution or ppt. obtained	NH_4^+confirmed.

$(NH_4)_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2NH_3 + 2H_2O$

$NH_3 + HCl \rightarrow NH_4Cl$

$2K_2HgI_4 + NH_3 + 3KOH \rightarrow HgO.Hg(NH_2)I + 7KI + 2H_2O$

Basic mercury (II)
amido-iodine(Brown precipitate)

FIRST GROUP OF BASIC RADICALS

The basic radicals of this group are Pb^{2+}& Ag^+. The group reagent is dilute HCl.

EXPERIMENT	OBSERVATION	INFERENCEE
(1) Original solution + Dilute HCl Filter and add water to ppt. and heat.	White ppt. is formed.	May be Pb^{2+}
	(a) ppt. dissolves in hot water.	May be Pb^{2+}
(2) Original solution + Potassium Chromate	(a) A yellow ppt. is formed	Pb^{2+} confirmed

(3) Original solution + KI solution ppt. obtained + water and heat it	A yellow ppt. soluble in hot water, on cooling reappears as golden yellow.	Pb^{2+} confirmed

$PbCl_2 + 2KI \rightarrow PbI_2 + 2KCl$
Yellow precipitate

$PbCl_2 + K_2CrO_4 \rightarrow PbCrO_4 + 2KCl$
Lead chromate
(Yellow precipitate)

SECOND GROUP OF BASIC RADICALS

The basic radicals of this group are May be Pb^{2+}, Hg^{2+}, Cu^{2+} & Cd^{2+}.The group reagent is dilute HCl+ H_2S gas.

EXPERIMENT	OBSERVATION	INFERENCEE
Original solution + dilute HCl+ H_2S gas.	A black ppt. is observed.	Pb^{2+}, As^{3+} and Cu^{2+}may be present

The identification of Pb^{2+}, As^{3+} and Cu^{2+} is as follows:

EXPERIMENT	OBSERVATION	INFERENCEE
(a) Original solution + Potassium Chromate	A yellow ppt. is observed	Pb^{2+} confirmed
(b) Original solution + NH_4OH	A bluish white ppt. soluble in excess of NH_4OH is observed which gives deep blue solution.	Cu^{2+}may be present
(c) Original solution + dil. acetic acid + Potassium ferrocyanide solution	A Chocolate red ppt. is observed	Cu^{2+} confirmed

Test for Lead ion (Pb^{2+})

$Pb^{2+} + CrO_4^{2-} \rightarrow PbCrO_4$
Lead chromate
$Pb^{2+} + 2I^- \rightarrow PbI_2$
Lead iodide(Yellow precipitate)

Test for Copper ion (Cu^{2+})

$2Cu^{2+} + SO_4^{2-} + 2NH_3 + 2H_2O \rightarrow Cu(OH)_2.CuSO_4 + 2NH_3$
$Cu(OH)_2.CuSO_4 + 8NH_3 \rightarrow 2\ [Cu(NH_3)_4]SO_4 + 2OH^- + SO_4^{2-}$
Tetraamminecopper (II)sulphate (Deep blue)

$[Cu(NH_3)_4] SO_4 + 4CH_3COOH \rightarrow CuSO_4 + 4CH_3COONH_4$
$2CuSO_4 + K_4[Fe(CN)_6] \rightarrow Cu_2[Fe(CN)_6] \downarrow + 2K_2SO_4$
(Chocolate brown precipitate)

THIRD GROUP OF BASIC RADICALS

The basic radicals of this group are Al^{3+} and Fe^{3+}. The group reagent is $NH_4Cl + NH_4OH$

EXPERIMENT	OBSERVATION	INFERENCE
(1) Original solution + $NH_4Cl(s) + NH_4OH$ in excess	(a) A white gelatinous ppt. is obtained	May be Al^{3+}
	(c) A reddish brown ppt. is obtained	May be Fe^{3+}
(2) Original solution + NaOH solution	(a) White gelatinous ppt. soluble in excess of NaOH is obtained which gives a colorless sol.	The basic radical is Al^{3+}
	(b) Dirty green ppt. insoluble in excess of NaOH	The basic radical is Fe^{2+}
	(c) Reddish brown ppt. insoluble in excess of NaOH	The basic radical is Fe^{3+}
(3) Clear solution of 2(a) + solid NH_4Cl and heat	The white gelatinous ppt. reappears	Al^{3+} confirmed
(4) Original solution + acidified $KMnO_4$ Solution which is added drop wise.	(a) The pink colour of $KMnO_4$ is not discharged.	Fe^{3+} confirmed.

Test for Aluminium ions (Al^{3+})

$AlCl_3 + 3NaOH \rightarrow Al(OH)_3 \downarrow + 3NaCl$
$Al(OH)_3 + NaOH \rightarrow NaAlO_2 \downarrow + 2H_2O$
White gelatinousprecipitate

FOURTH GROUP OF BASIC RADICALS

The basic radicals of this group are Zn^{2+}, Mn^{2+}, Co^{2+} & Ni^{2+}. The group reagent is $NH_4Cl + NH_4OH$ and H_2S (g)

EXPERIMENT	OBSERVATION	INFERENCE
(1) Original solution + $NH_4Cl(s)$ + NH_4OH in excess + $H_2S(g)$	(a) a white ppt. is obtained	May be Zn^{2+}, Mn^{2+} or Co^{2+}

	(b) Buff or pale pink or flash colored ppt. soluble in dilute HCl is obtained.	May be Mn^{2+}
(2) Original solution + NaOH solution	(a) The white ppt. is soluble in excess of NaOH giving a colorless solution.	Zn^{2+} confirmed
	b)The white ppt. insoluble in excess of NaOH but turns brown	Mn^{2+} confirmed
(3)Dissolve the 1(a) in aqua-regia, evaporate to dryness, add 1 ml of distilled water and divide the resultant product into two parts. Part (1) + 1 ml amyl alcohol + 100 mg solid NH_4SCN & stir it. Part (2) + 5 drops dimethyl glyoxime reagent + NH_4OH.	 A blue colour in alcohol layer A pink ppt.	 Co^{2+}confirmed Ni^{2+} confirmed

Test for Zinc ion (Zn^{2+})

$ZnS + 2HCl \rightarrow ZnCl_2 + H_2S$
$ZnCl_2 + 2NaOH \rightarrow Zn(OH)_2 + 2NaCl$
$Zn(OH)_2 + 2NaOH \rightarrow Na_2ZnO_2 + 2H_2O$

Test for Manganese ion (Mn^{2+})

$MnS + 2HCl \rightarrow MnCl_2 + H_2S$
$MnCl_2 + 2NaOH \rightarrow Mn(OH)_2 + 2NaCl$
(White precipitate)
$Mn(OH)_2 + [O] \rightarrow MnO(OH)_2$
(Brown colour)

Test for Nickel ion (Ni^{2+})

$3NiS + 2HNO_3 + 6HCl \rightarrow 3NiCl_2 + 2NO + 3S + 4H_2O$

Test for Cobalt ion (Co^{2+})

$CoS + HNO_3 + 3HCl \rightarrow CoCl_2 + NOCl + S + 2H_2O$
$CoCl_2 + 7KNO_2 + 2CH_3COOH \rightarrow K_3 [Co(NO_2)_6] + 2KCl + 2CH_3COOK + NO + H_2O$
Potassium
hexanitritocobaltate(III)
(Yellow precipitate)

FIFTH GROUP OF BASIC RADICALS

EXPERIMENT	OBSERVATION	INFERENCE
Original solution + Potassium Chromate	A yellow ppt.	Ba^{2+}confirmed.
To O.S. add ammonium sulphate solution. Heat and scratch the sides of the test tube with a glass rod and cool.	A White ppt.	Sr^{2+}confirmed
To O.S. add ammonium oxalate solution and shake well.	A white ppt	Ca^{2+}confirmed

Test for Barium ion (Ba^{2+})

$BaCO_3 + 2CH_3COOH \rightarrow (CH_3COO)_2Ba + H_2O + CO_2$
$(CH_3COO)_2Ba + K_2CrO_4 \rightarrow BaCrO_4 + 2CH_3COOK$
Barium chromate(yellow precipitate)

Test for Strontium ion (Sr^{2+})

$SrCO_3 + 2CH_3COOH \rightarrow (CH_3COO)_2Sr + H_2O + CO_2$
$(CH_3COO)_2Sr + (NH_4)_2SO_4 \rightarrow SrSO_4 + 2CH_3COONH_4$
Strontium
sulphate(White precipitate)

Test for Calcium ion (Ca^{2+})

$CaCO_3 + 2CH_3COOH \rightarrow (CH_3COO)_2Ca + H_2O + CO_2$
$(CH_3COO)_2Ca + (NH_4)_2C_2O_4 \rightarrow (COO)_2Ca + 2CH_3COONH_4$
(White precipitate)

SIXTH GROUP OF BASIC RADICALS

EXPERIMENT	OBSERVATION	INFERENCE
Original solution + excess NH_4OH + ammonium hydrogen phosphate	A white ppt.	Mg^{2+} confirmed.

Test for Magnesium ion (Mg^{2+})

$Mg^{2+} + Na_2HPO_4 \rightarrow Mg(NH_4)PO_4 + NH_4OH + 2Na^+ + H_2O$
(White precipitate)

*Sometimes precipitate of magnesium ammonium phosphate appear s after some time. So warm the solution and scratch the sides of test tube after adding sodium hydrogen Phosphate solution.

Experiment:

Aim: Demonstration of Le Chatelier's principle through a reversible chemical reaction involving a hydrated Cobalt complex.

Materials Pipets, Beral-type, 3 Silver nitrate solution, AgNO3, 0.1 M, 3 mL Test tube rack Water, distilled Test tubes, borosilicate glass, medium-size Beakers, 400-mL, 2 (approximately 19 × 150 mm), 5

Requirement:

Apparatus: Hot plate, test tube, Beaker, glass rod, dropper and pH paper.

Chemicals: Cobalt(II) chloride solution, $CoCl_2$, 0.1 M, conc. HCl, $AgNO_3$ 0.1 M, distilled water

Theory: Demonstrate the power of balance in a reversible chemical reaction by showing how chemical equilibrium represents a true chemical balancing act and thus responds dramatically to anything that might upset that balance.

Chemical equilibrium is a dynamic condition. At equilibrium the concentrations of reactants and products remain unchanged. It is the ratio of product to reactant concentrations, governed by the stoichiometry of the balanced chemical equation, that is constant. The concentrations of individual reactants and products are affected by changes in the other terms in the equilibrium constant ratio or expression. And the equilibrium "constant" itself is temperature dependent. The effect of concentration, temperature, and pressure changes on the position of chemical equilibrium for a reversible chemical reaction is expressed intuitively in LeChâtelier's Principle: "*If the conditions of a system, initially at equilibrium, are changed, the equilibrium will shift in such a direction as to tend to restore the original conditions*." The chemical reaction demonstrated herein involves the formation of complex ions between $Co2^+$ and water molecules or chloride ions, respectively.

$$\underset{\text{pink}}{[Co(H_2O)_6]^{2+}} + 4Cl^- + \text{heat} \longleftrightarrow \underset{\text{blue}}{CoCl_4^{\ 2-}} + 6H_2O \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ eq 1}$$

A solution of cobalt(II) ion in water is pink, the color of the complex ion formed between Co2+ ions and water molecules. When chloride ion in the form of hydrochloric acid is added to the solution, the color changes to blue, corresponding to the formation of a charged coordination complex between Co^{2+} and chloride ions. This reaction is reversible and quickly reaches a position of chemical equilibrium, which is immediately evident by the color of the solution. In terms of the position of equilibrium for this reaction, addition of Cl^- ion (excess reactant) shifts the equilibrium to the right (toward $CoCl_4^{2-}$ formation) to consume some of the added reactant and thus restore the equilibrium condition. If the blue solution corresponding to $CoCl_4^{2-}$ is diluted by the addition of water (a product of the above reaction), the effect is to shift the equilibrium back to the left, toward $[Co(H_2O)_6]^{2+}$. This observation requires a slightly different explanation, since technically the concentration of water (solvent) in an aqueous solution is constant. The effect can be explained in terms of the equilibrium constant for the reaction.

Addition of $AgNO_3$ to the blue solution of $CoCl_4^{2-}$ results in the formation of a copious white precipitate of AgCl, via the reaction

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl\downarrow(s)$$

and a pink solution of $[Co(H_2O)_6]^{2+}$. Depletion of the chloride ion concentration due to the formation of insoluble AgCl shifts the equilibrium in Equation 1 back to the left, toward reactant formation, in order to offset the effect of this change. The effect of heat is explained by noting that reaction (1) is endothermic, so that heat may be thought of as a reactant in the reaction equation. Addition of excess reactant in the form of heat shifts the equilibrium in the direction in which heat is absorbed in order to "consume" the excess reactant. Adding heat shifts the reaction in Eq (1) to the right (blue), while removing heat shifts it back to the left (pink).

Preparation

1. Obtain four medium-sized test tubes and label them: P1, P2, B1, and B2.

2. Measure approximately 5 mL of 0.1 M $CoCl_2$ solution into each of the four test tubes.

3. Slowly and carefully add 5 mL of concentrated HCl to test tubes B1 and B2.

Note: The solution in test tubes B1 and B2 should turn blue.

4. Set aside test tubes P1 and B1 as control solutions.

5. Obtain a 400-mL beaker and fill it with tap water about half full. Use a hot plate to heat the water to 80–85 °C.

6. Obtain a second 400-mL beaker and prepare an ice water bath filled about half full.

Procedure

1. Add 5 mL of concentrated HCl to test tube P2 in approximately 0.5 mL increments until the solution turns blue in color.
2. Once the solution has turned blue in test tube P2 add 5 mL of distilled water in approximately 0.5 mL increments until the solution reverts to its original pink color.

3. Add 2 mL of 0.1 M $AgNO_3$ solution to test tube B2. Note: A large amount of white precipitate will form while the supernatant will be pink.

4. Obtain test tube P2, which contains roughly 15 mL of pink solution, and place it in the hot water bath. The solution will gradually change in color from pink to lavender blue.

5. Using a test tube clamp, remove test tube P2 from the hot water bath and immediately immerse it in the ice water bath. The solution should revert to pink.

Experiment:

Aim: To demonstration of endothermic and exothermic reactions.

Requirement:

Apparatus: Test tube, beaker, glass rod, dropper and thermometer.

Chemicals: Citric acid, $NaHCO_3$, HCl, Mg strips, distilled water

Introduction

Chemical reactions that release energy are called exothermic reactions. Some chemical reactions absorb energy and are called endothermic reactions. Here we study one exothermic and one endothermic reaction in this experiment.

Reaction I: The temperature change in the reaction between citric acid solution and baking soda will be studied. An equation for the reaction is:

$$H_3C_6H_5O_7(aq) + 3\ NaHCO_3(s) \rightarrow 3\ CO_2(g) + 3\ H_2O(l) + Na_3C_6H_5O_7\ (aq)$$

Reaction II: The reaction between magnesium metal and hydrochloric acid. An equation for this reaction is:

$$Mg(s) + 2\ HCl(aq) \rightarrow H_2(g) + MgCl_2(aq)$$

Procedure: 1) Take 15 mL of citric acid solution in a 50 mL and add 8 mL saturated solution of sodium bicarbonate stir it slowly and record initial and final minimum temperature (during evolution of CO_2).

2) Take 15 mL of dil. HCl in a beaker and add slowly Mg (0.5 mg), evolution of H_2 will be observed record the initial and final temperature.
CAUTION: Do not breathe the vapors! Collect data until a maximum temperature has been reached and the temperature readings begin to decrease.

Observation table

	For Citric acid	**For Mg**
Initial temperature t_1		
Final temperature t_2		
Temperature change ($\Delta t = t_2 - t_1$)		

Calculate the temperature change (Δt) for both the reactions =

- Explain out of these two reactions which one is exothermic and which is endothermic.